SNS COLLEGE OF ENGINEERING

SARAVANAMPATTI, COIMBATORE – 641 035.

DEPARTMENT OF CIVIL ENGINEERING

ENVIRONMENTAL ENGINEERING LABORATORY

USER MANUAL

Environmental Engineering Laboratory

Approved By:

Faculty In-charge	HOD / CIVIL	PRINCIPAL

Department of Civil Engineering – SNS College of Engineering

BONAFIDE CERTIFICATE

Registration No: | | | | | | | | | | | | |

Certified that this is the bonafide record of work done by Mr / Ms _____ of _____ semester B.E. Civil Engineering during the academic year _____ in the Environmental Engineering laboratory.

HEAD OF THE DEPARTMENT **STAFF In-charge**

Submitted for the university practical examination held on _____ at Environmental Engineering laboratory in SNS College of Engineering, Coimbatore.

Internal Examiner **External Examiner**

Date: **Date:**

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Code: CE2356	Environmental Engineering Laboratory	L 0	T 0	P 3	C 2

Anna University Syllabus / Regulation 2008

OBJECTIVE

This subject includes the list of experiments to be conducted for characterisation of water and municipal sewage. At the end of the course, the student is expected to be aware of the procedure for quantifying quality parameters for water and sewage.

TOTAL: 45 PERIODS

REFERENCES

1. Standard methods for the examination of water and wastewater, APHA, 20th Edition, Washington, 1998.
2. Garg, S.K., "Environmental Engineering Vol. I & II", Khanna Publishers, New Delhi.
3. Modi, P.N., "Environmental Engineering Vol. I & II", Standard Book House, Delhi-6.

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Exp No: 1 Date:	Sampling and preservation methods and significance of characterisation of water and wastewater

Objective:

By this experimental study, proper sampling and preservation techniques are studied in order to maintain the integrity of the sample. Improper sample handling can invalidate the results of any analysis that a laboratory performs.

Aim:

To be conversant in sampling and preservation methods and significance of characterisation of water and wastewater.

Theory:

Introduction

In this lesson we will be considering **sampling**, which is the process used to select a small portion of water or wastewater for the purpose of determining the characteristics of an entire batch of water or wastewater. Sampling is used every day at water and wastewater treatment plants to determine the characteristics of the water. Sampling may be used to test the finished water for regulatory purposes - to ensure that the treatment plant is treating the water in compliance with regulations. Or sampling may be used to help the operator tweak the chemical feed rates to make the treatment procedure as efficient as possible.

You will use sampling in every lab exercise completed in this course. For example, in the pH lab you'll take a small sample of water and analyze the pH of that sample, then assume that the pH of all of the water at a certain stage in the treatment process has the same pH value as the sample tested. This is just one of many examples of sampling which you will be exposed to as a treatment plant operator. Since the value of any laboratory result depends on the quality of the sample taken, bad sampling can lead to laboratory results which are misleading and worse than no results at all.

WATER SAMPLING

The significance of a chemical analysis depends to a large extent on the sampling programme. An ideal sample should be done which is both valid and representative. These conditions are met by collection of samples through a process of random selection. This ensures that the composition of the sample is identical to that of the water body from which it is collected and the sample shares the same physico-chemical

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characteristics with the sampled water at the time and site of sampling. The relevant factors for any sampling programme are (a) frequency of sample collection, (b) total number of samples, (c) size of each sample, (d) sites of sample collection, (e) method of sample collection, (f) data to be collected with each sample, and (g) transportation and care of samples.

For analysis of natural and waste water, two principal types of sampling Procedures are employed:

SPOT OR GRAB SAMPLES

These are discrete portions of water samples taken at a given time. A series of grab samples, collected from different depths at a given site, reflect variations in constituents over a period of time. The total number of grab samples should satisfy the requirements of the sampling programme.

COMPOSITE SAMPLES

These are essentially weighted series of grab samples, the volume of each being proportional to the rate of flow of the water stream at the time and site of sample collection. Samples may Composed over anytime period, such as 4, 8 or 24 hours, on the purpose of analysis. Such composite samples are useful for determining the average condition which , when correlated with flow, , can be used for computing the material balance of a stream of water body over a period of time. It may be stated in general, that it is more meaningful to analyse a large number of separate samples taken at different times and different locations than to *compile* and analyse a *single* representative sample. Separate samples must be collected for chemical and biological analysis. , since the sampling and preservation techniques are quite different. For accurate analysis, *it* is desirable to allow a short-time interval between sampling and analysis. As matter of fact temperature, pH and dissolved gases (D.O.) must be determined in the field and as quickly as possible after sampling. Collection of truly representative sample is as important as sample preservation. A representative *single* sample *is* taken from a number of different locations over a long *period* of *time.* In general, it is more significant to analyse a large number of separate samples taken at different times and different locations than it is to compile and analyse a single representative sample. It is essential to keep accurate records of the location, time and conditions of sample collection.

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PRESERVATION

It is essential to protect samples from changes in composition and deterioration with aging due to various interactions. The optimum sample holding time ranges from zero for parameters, such as pH, temperature and D.O to one week for metals. The preservation techniques for various parameters are summarized in the following table. As mentioned above, these are essential for retarding biological action, hydrolysis of chemical compounds and complexes and reduction of volatility of constituents. It is desirable for accurate results that analysis must be undertaken within 4 hours for some parameters and 24 hours for others, from the time of collection and it must be concluded within a week.

WATER SAMPLE PRESERVATION

Parameter	Min Sample (ml)	Container type	Preservation
pH	100	Polythene	Measure within 0-4 hours
DO	100	Polythene	
COD	500	Polythene	Add H_2SO_4 to pH; refrigerate
Nitrogen Ammonia	500	Polythene	Analyse as soon as possible, add 0.8ml concentration of H_2SO_4 / L
Nitrate + Nitrite	500	Polythene	Add 40 mg $HgCl_2$ / L and refrigerate
Cyanide	500	Polythene	Add NaOH to pH 12 and 25 ml of 2% ascorbic acid and refrigerate
Sulphide	500	Polythene	Add 1ml of 2N $Zn(CH_3COO_2)$ and 2ml of 1M NaOH stir and refrigerate
Phosphate	500	Polythene / Glass	Add 40mg $HgCl_2$ / L and refrigerate
Phenol	500	Polythene / Glass	Acidify with H_3PO_4 to pH 0.4 and add 1g Cu_2SO_4 $_5H_2O$ per L to inhibit biodegradation
Tannin and Lignin	500	Polythene / Glass	Analyse as soon as possible
Chromium, Arsenic, Lead, Zinc & Mercury	500	Polythene / Glass	Add 5ml Concentration HNO_3 / L and refrigerate
E.Coli / Total bacteria / Actenomycetis	100	Glass bottle	Sterilize the bottles in auto clave at 121^o C at 15 lb/1nch^2 pressure for 15 minutes, collect the sample in sterilize bottle and refrigerate immediately.
Microplankton /	500	Glass bottle	Add 5ml formalin per 100ml sample

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Algae and other biological organisms			and refrigerate immediately.

Comment:

The detailed report of sampling and preservation methods and significance of characterisation of water and wastewater is studied.

Inference:

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Possible Viva-Voce

S.No	Query
1	Define pH and its range in potable water.
2	Explain the types of samples.
3	Define BOD & COD.
4	How the water does turn acidic or basic?
5	What is the pH of rain water?
6	Which types of solids are found in polluted water?
7	Name few micro-organisms present in water sample?
8	What happens when acid is poured in water?
9	Define potable water?
10	List out the characteristics of water?

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Exp No: 2 Date:	Determination of pH

Objective:

By this experimental procedure, significance of pH and its effect on water sample is perceived.

Aim:

To determine the pH of the given sample using pH meter.

Theory:

pH of aqueous solutions can be defined as negative logarithm of hydrogen ion concentration. pH values ranging from 0 to 7 are acidic, and from 7 to 14 are alkaline. The effect of pH on the chemical and biological properties of liquids makes its determination very important. It is used in several calculations in analytical work. The pH determination is usually done by electrometric method which is the most accurate method and free of interference. pH is measured by a pH meter using a glass electrode which generates a potential varying linearly with pH of solution in which it is immersed. The basic principle of electrometric pH measured is determination of hydrogen ion by a standard hydrogen electrode and a reference electrode.

Apparatus Required:

1) pH meter
2) Water samples

Procedure:

- The protective cover of the pH meter is removed and pH meter is turned on by the switch.
- Rinse the electrode thoroughly with distilled water.
- Dry electrode by gently blotting with a soft tissue paper and standardize instrument with electrode immersed in a buffer solution.
- Remove the electrode from buffer, rinse thoroughly and blot with a soft tissue paper to dry the electrode.
- Immerse in a second buffer below pH 10, approximately 3 pH units different from the first, the reading should be within 0.1 unit for the pH of the second buffer.
- Rinse the electrode thoroughly, blot and dry and determine the pH of unknown sample.

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- Dip the pH meter in the sample solution for about one minute.
- The reading is taken after the indicated value remains constant for a minute.

Table contains the pH unit of different samples

S.No	Description of the sample	Temperature of the sample (°C)	pH Unit	Remarks
1	Sample A			
2	Sample B			
3	Sample C			
4	Sample D			

Result:

The permissible pH limit for drinking water is 6.5 – 8.6. The given sample is suitable / not suitable for drinking purpose.

The pH values of the given samples are

1. Sample A – 0 – Acidic / Basic
2. Sample B – 0 – Acidic / Basic
3. Sample C – 0 – Acidic / Basic
4. Sample D – 0 – Acidic / Basic

Inference:

Environmental Engineering Laboratory

Possible Viva-Voce

S.No	Query
1	pH is defined as?
2	pH of neutral water is?
3	The acceptable value of pH of potable water is?
4	The inner solution present in the glass electrode of Ph meter is?
5	Is it possible to store the buffer solution for a minimum period at room temperature?
6	Define pH scale?
7	Possible reason for a relatively low pH value in a river water sample is due to?
8	Possible reason for a relatively high pH value in a river water sample is due to?
9	How to reduce the level of pH?
10	How to increase the level of pH?

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Exp No: 3 Date:	Determination of Turbidity

Objective:

By this experimental procedure, the effect of turbidity on water quality and also the importance of determining the turbidity level in water is examined.

Aim:

To determine the turbidity of the given sample using turbidity meter.

Theory:

Turbidity of water is caused by suspended particles, primarily of clay, silt, organic matter, and microorganisms. In natural waters, turbidity can range from a few NTU to about 1,500 NTU. While turbidity is not harmful by itself, excess turbidity hinders the efficiency of disinfection which can result in unsafe drinking water. As a result, the EPA Drinking Water Standards limit turbidity to 1 NTU in filtered water. In practice, coagulation, flocculation, and sedimentation followed by effective filtration should result in effluent water containing less than 0.2 NTU. The test for turbidity is a physical test rather than a chemical test. An instrument called a nephelometer is used to measure the amount of light which is scattered by hitting turbidity particles in the water. In the treatment plant, operators usually test the turbidity of the finished water at intervals throughout the day. This gives the operator an indication of how well the water is being treated. If the effluent water has an unexpectedly high turbidity, then the chemical feed rates may need to be adjusted or the filters may need to be backwashed.

Apparatus required:

1) Turbidity meter
2) Sample cells
3) Standard flask
4) Funnel
5) Wash bottle
6) Tissue papers

Chemicals required:

1) Hexamethylenetetramine
2) Hydrazine sulphate
3) Distilled water

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Procedure:

For testing the given sample first the reagents are to be prepared. Then the turbidity meter is required to be calibrated.

Preparation of Reagents

a) **Hydrazine sulphate**

- Weigh accurately 1g of hydrazine sulphate and dissolve it in turbidity free distilled water.
- Take 100ml standard measuring flask and place a funnel over it.
- Transfer it to a 100ml standard flask and make up to 100 ml using turbidity free distilled water.

b) **Hexamethylenetetramine**

- Weigh accurately 10g of hexamethylenetetramine and dissolve it in turbidity free distilled water.
- Take 100ml standard measuring flask and place a funnel over it.
- Transfer it to a 100ml standard flask and make up to 100 ml using turbidity free distilled water.

c) **Standard 4000 NTU Solution**

- Mix 5ml of hydrazine sulphate solution and 5ml of hexamethylenetetramine solution in a 100ml standard measuring flask.
- Allow the mixture to stand for 24 hours.
- After 24 hours, make up the volume to 100ml using turbidity free distilled water.
- The standard 4000 NTU solution is ready.

Calibration of Turbidity meter

- Using the standard solution calibrate the instrument.
- The instrument is having four knobs, out of which the two knobs in the bottom is the set zero knob, this is for setting the instrument to zero.
- The one which is there in the top left hand side is the calibration knob, used for the calibration.
- The other one in the top is the knob for setting the detection range. It is adjusted to 1000 NTU range.

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Step 1

- To the sample cell, add turbidity free distilled water up to the horizontal mark, wipe gently with the soft tissue. Place it in turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell. Now using the set zero knob, adjust the reading to zero.

Step 2

- According to our need, prepare a standard solution. In this case, a 200 NTU solution is prepared by diluting the standard 4000 NTU solution and added to the sample cells, up to the horizontal mark, wipe gently with soft tissue. Place it in turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell.

- If the instrument is not showing 200 NTU, using the calibration knob adjust the reading to 200 NTU.
- Repeat the procedure for two / three times.
- Now the instrument is calibrated.

Testing of water sample

- To the sample cell, add turbidity free distilled water up to the horizontal mark, wipe gently with the soft tissue. Place it in turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell.
- Check for the reading in the turbidity meter. Wait until you get a stable reading.
- Now note down the value displayed as the turbidity of the water sample placed in the instrument.

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Table contains the turbidity unit of different samples

S.No	Description of the sample	Temperature of the sample (°C)	Turbidity (NTU)	Remarks
1	Sample A			
2	Sample B			
3	Sample C			
4	Sample D			

Result:

The turbidity of the given sample is determined.

Sample A –

Sample B –

Sample C –

Sample D –

Inference:

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Possible Viva-Voce

S.No	Query
1	Turbidity is caused by?
2	The turbidity is measured based on?
3	Does colour of water sample affects the turbidity?
4	In a nephelo turbidity meter the light detectors are at?
5	What is the unit of turbidity?
6	What is the light source for the nephelo turbidity meter?
7	Does aquatic life affects the turbidity in water?
8	The standard unit of turbidity is considered as that produced by?
9	The material used in the standard solution for nephelometer is?
10	Mixture of hydrazine sulphate and hexamethylenetetramine solution is allowed to stand for?

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Exp No: 4 Date:	Determination of Hardness

Objective:

By this experimental procedure, the suitability of water for different purpose is examined based on the type of hardness.

Aim:

To determine the total hardness of the given sample.

Theory:

Water that has high mineral content is known as Hard water. Hard water contains bicarbonate, chlorides and sulphates of calcium and magnesium. When treated hard water with soap, it gets precipitated in the form of insoluble salts of calcium and magnesium. Hardness of water is a measure of the total concentration of the calcium and magnesium ions expressed as calcium carbonate. There are two types of hardness,

1) Temporary hardness
2) Permanent hardness

Temporary hardness is due to the presence of bicarbonates of calcium and magnesium. It can be easily removed by boiling.

Permanent hardness is due to the presence of chlorides and sulphates of calcium and magnesium. This type of hardness cannot be removed by boiling.

A water sample is buffered to pH 10.1 and taken into a conical flask. If indicators die like EBT, when added to a solution containing calcium and magnesium ions, the colour of the solution turns to wine red. EDTA, the titrant, complexes with magnesium and calcium ions, removing them from association with the indicator. When all the Mg and Ca are complexed with EDTA, the indicator will turn blue. This is the end point of titration.

Apparatus Required:

1) Burette with Burette stand and porcelain tile
2) Pipette with elongated tips
3) Pipette bulb
4) Conical flask
5) Measuring jar
6) Wash bottle
7) Beaker

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Chemical required:

1) Ammonium chloride
2) Ammonium hydroxide
3) EDTA (Disodium salt of EDTA)
4) Erichrome Black T (EBT)
5) Magnesium sulphate

Procedure:

Preparation of reagents

Buffer solution preparation

- Switch on the electronic balance, keep the weighing pan and set the reading to zero.
- Measure 50ml of distilled water and transfer it to beaker.
- Weigh 1.179g of EDTA, now the weight is 1.179 gms.
- Transfer the content to the beaker having 50ml of distilled water and dissolve it thoroughly.
- Weigh 16.9g of Ammonium chloride.
- Add it to the contents in the beaker and dissolve it thoroughly.
- Weigh 780mg of Magnesium sulphate and transfer it to the beaker.
- Measure 143ml of Ammonium hydroxide solution using measuring cylinder and add it to the contents in the beaker.
- Place the funnel over the 250ml standard flask and transfer the dissolved contents from beaker.
- Make the volume upto 250ml mark by adding distilled water. Transfer the buffer solution to a clean reagent bottle labelled as buffer solution. This buffer solution is used to maintain the pH of water sample between 9 and 10.

Erichrome Black T

- Weigh 0.5g of EBT.
- Transfer it to 100ml standard flask using funnel.
- Add distilled water in the standard flask and make the volume exactly upto 100ml mark.
- Put the lid and shake the content well.
- Transfer the solution to a clean reagent bottle named EBT.

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Standard EDTA solution (0.02M)

- Switch on the electronic balance, keep the weighing pan and set the reading to zero.
- Weigh 3.723g of EDTA sodium salt.
- Transfer the entire content to 1000ml standard flask.
- Fill with distilled water up to 1000ml mark.
- Put the lid and shake the content well.
- For easy handling take the EDTA solution in a 250ml beaker.

Total hardness test

- Pipette 20ml of hard water sample and transfer it to a clean 250ml conical flask.
- Add 2ml of Ammonia buffer solution to the water sample so that the pH will be maintained between 9 and 10.
- Add few drops of EBT indicator to the conical flask and the sample turns to wine red in colour.
- Before starting the titration rinse the burette with few ml of EDTA. Fill the burette with EDTA solution and adjust to zero then fix it in burette stand.
- Titrate the sample against the EDTA solution in the burette till all calcium and magnesium ions present in the samples reacts with the EDTA. The appearance of blue colour indicates that all Ca & Mg ions are complexed with EDTA and forms a metal EDTA complex i.e. the end point of the titration.
- Note down the burette reading.
- Repeat the titration for concordant values.

Tabulation:

Burette solution: EDTA

Pipette solution: Sample

Indicator: EBT

End point: Appearance of blue colour

Titration I – Estimation of total hardness

Burette solution: EDTA

Pipette solution: Normal water

Additional solution: 5ml of buffer solution

Indicator: EBT

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End point: Appearance of blue colour

S.No	Volume of sample (ml)	Burette reading		Volume of EDTA consumed (ml)
		Initial (ml)	Final (ml)	
1				
2				
3				
			Concordant Value	

Volume of EDTA =

Normality of EDTA =

Volume of Sample =

Equivalent weight of $CaCo_3$ =

$$\text{Total hardness} = \frac{\text{Volume of EDTA} \times N \times 50 \times 1000}{\text{Volume of Sample taken}}$$

Calcium Hardness as $CaCo_3$ equivalent (mg/l) = mg/l as $CaCo_3$ equivalent

Result:

The amount of total hardness present in the given sample is _____

Inference:

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Possible Viva-Voce

S.No	Query
1	The cation that cannot cause hardness is?
2	Addition of complexometrically neutral Mg to buffer is to?
3	The hard water does not lather with soap because soap is precipitated?
4	The buffer can be stored for?
5	The need of buffer in titration is for?
6	Does hard water consumes more soap for cleaning purpose?
7	EDTA means?
8	The hard water is?
9	Buffer solution is the solution which resists change in its?
10	The Erichrome Black T is used as a?

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Exp No: 5 Date:	Determination of Chlorides

Objective:

By this experimental procedure, the level of chlorine is revealed which is useful in determining suitable water supply schemes, types of desalting apparatus and to control pumping of ground water when seawater intrusion is found.

Aim:

To estimate amount of chloride present in the given sample of water.

Theory:

Chlorides are widely distributed as salts of calcium, sodium and potassium in water and waste water. The major taste producing salts in water are sodium chloride and calcium chloride. The salty taste is due to chloride anions and association cations in water. In some water which is having only 250 mg/l of chloride may have a detectable salty taste if the cat-ion present in the water is sodium. On the other hand, a typical salty taste may be absent if the water is having very high chloride concentration for example 1000 mg/l. This is because the predominant cation present in the water is not sodium but either calcium or magnesium may be present. The amount of chloride present in water can be easily determined by titrating the given water sample with silver nitrate solution. The silver nitrate reacts with chloride ion according to 1 mole of $AgNO_3$ reacts with 1 mole of chloride. The titrant concentration is generally 0.02M. Silver chloride is precipitated quantitatively, before red silver chromate is formed. The end of titration is indicated by formation of red silver chromate from excess silver nitrate. The results are expressed in mg/l of chloride (Cl^- with a molecular weight of 35.453 g/mol).

Apparatus Required:

1) Burette with Burette stand and porcelain tile
2) Pipette with elongated tips
3) Pipette bulb
4) Conical flask
5) Wash bottle
6) Beaker

Chemical required:

1) Silver nitrate

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2) Phenolphthalein indicator

3) Sodium chloride

4) Potassium chromate

Procedure:

Preparation of reagents

Standard Sodium Chloride Solution

- Switch on the electronic balance, keep the weighing pan and set the reading to zero.
- Weigh 1.648g of Sodium Chloride
- Transfer the contents to the beaker containing distilled water. Using glass rod, dissolve the contents thoroughly.
- Transfer the contents in the beaker to a 100ml standard flask; fill distilled water up to 100ml mark.
- Transfer it to 100ml standard flask using funnel.

Standard Silver Nitrate (0.0282N)

- Weigh 4.791g of silver nitrate and transfer it to the beaker with distilled water.
- Transfer the contents in the beaker to a 100ml standard flask; fill distilled water up to 100ml mark.
- Standardize it against 0.0282N NaCl solution. Store it in a amber bottle.

Potassium Chromate indicator

- Weigh 25g of Potassium Chromate. Transfer it to the beaker contains distilled water. Add few drops of Silver Nitrate solution until slight red precipitate is formed.
- Allow it to stand for 12 hours. After 12 hours filter the solution using filter paper and dilute the filtrate to 1000ml using distilled water.

Testing of water sample

- Before starting the titration rinse the burette with silver nitrate solution. Fill the burette with silver nitrate solution of 0.0282N. Adjust to zero and fix the burette in stand.
- Take 20ml of the sample in a clean 250ml conical flask.
- Add 1ml of Potassium Chromate indicator to get light yellow colour.
- Titrate the sample against silver nitrate solution until the colour changes from yellow to brick red i.e. the end point.
- Note the volume of silver nitrate added (A).

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- Repeat the procedure for concordant value.

Blank Titration

- Take 20ml of the distilled water sample in a clean 250ml conical flask.
- Add 1ml of Potassium Chromate indicator to get light yellow colour.
- Titrate the sample against silver nitrate solution until the colour changes from yellow to brick red i.e. the end point.
- Note the volume of silver nitrate added (B).
- Repeat the procedure for concordant value.

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Tabulation:

Titration – Chlorides

Burette solution: $AgNO_3$ (Silver Nitrate)

Pipette solution: Sample

Indicator: Potassium dichromate

End point: Appearance of Brick red colour

S.No	Volume of sample (ml)	Burette reading		Volume of $AgNO_3$ consumed (ml)
		Initial (ml)	Final (ml)	
1				
2				
Blank				
			Concordant Value	

Volume of Silver Nitrate for Sample (V_S) =

Volume of Silver Nitrate for Blank (V_B) =

Normality of Silver Nitrate =

Volume of Sample =

Equivalent weight of Chloride =

$$\text{Chlorides (mg/L)} = \frac{(V_S - V_B) * \text{Normality} * 35.45 * 1000}{\text{Volume of Sample taken}}$$

Chlorides (mg/L) = _____ mg/L

Result:

Amount of chloride present in the given distilled water sample is _____

Inference:

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Possible Viva-Voce

S.No	Query
1	The limit of chlorides in drinking water as per IS code is?
2	Why silver nitrate is stored in a brown bottle?
3	What is the colour of silver chromate?
4	When both hardness and chloride content are very high above 500mg/l, then the water will be?
5	Presence of chloride can corrode?
6	What is the chloride concentration in sewage?
7	What happens to the chloride consumed by human beings?
8	Chlorides give salty taste to water particularly when present as?
9	The point at which a clear visual change is observed after the reaction between titrant and titration is called?
10	Name the most common ion in water?

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Exp No: 6 Date:	Determination of Optimum coagulant dosage

Objective:

By this experimental procedure, the process of coagulation and flocculation using alum to remove turbidity of water is made clear.

Aim:

To determine the quantity of alum required to coagulate the given sample of raw water.

Theory:

The jar test is a method of measuring the effect of coagulation, flocculation, and sedimentation on turbidity. Coagulation/flocculation is the process of binding small particles in the water together into larger, heavier clumps which settle out relatively quickly. The larger particles are known as floc. Properly formed floc will settle out of water quickly in the sedimentation basin, removing the majority of the water's turbidity. In many plants, changing water characteristics require the operator to adjust coagulant dosages at intervals to achieve optimal coagulation. Different dosages of coagulants are tested using a jar test, which mimics the conditions found in the treatment plant. The first step of the jar test involves adding coagulant to the source water and mixing the water rapidly (as it would be mixed in the flash mix chamber) to completely dissolve the coagulant in the water. Then the water is mixed more slowly for a longer time period, mimicking the flocculation basin conditions and allowing the forming floc particles to cluster together. Finally, the mixer is stopped and the floc is allowed to settle out, as it would in the sedimentation basin. The amount of alum required for coagulant depends on the turbidity and colour of raw water. The optimum dosage of alum is usually found to vary from 5 mg/l for relatively clear water to about 85 mg/l for highly turbid water. The average normal dosage is about 17 mg/l.

Apparatus Required:

1) A stirring machine with paddles capable of variable speeds from 0 to 100 rpm.
2) Analytical balance
3) Beakers
4) Pipette
5) Watch clock

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Chemical required:

1) Coagulant and coagulant aids.

Procedure:

- Fill 1 litre water sample in each jar.
- Attach he sample jar to the stirring device by lifting the paddles in the right upward direction.
- Add coagulation solution in progressive volume of 0.25ml to 0.5ml, 0.75ml, 1ml, 1.25ml, 1.5ml into the series of the sample jars.
- Flocculate the sample rapidly for about one minute with mechanically operated paddles at 60 – 80 rpm followed by gentle stirring at about 30 rpm for 15 minutes.
- Remove the jars from the stirring device after stirring is completed.
- Let the sample (jars) be kept immovable for 30 minutes for settling of flocs.
- Select the minimum dosage giving the best floc and the most floc settled.
- This is the optimal coagulant dosage.

Tabulation:

S.No	Dosage of Coagulant (gm/l)	Depth of Sediment (cm)	Observation in regard to quantity of floc formed
1			
2			
3			
4			

Result:

Select optimum dosage of alum is _____

Inference:

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Possible Viva-Voce

S.No	Query
1	Chemical formula of Alum?
2	Why alum is preferred to other coagulants?
3	What is the difference between coagulation and flocculation?
4	What are coagulant aids?
5	What is the significance of pH in coagulation using alum?
6	What factors affect the sedimentation of a discrete particles settling in a quiescent liquid?
7	What is NTU?
8	1 PPM is equals to ___mg/l.
9	What are the advantages of using coagulant aids?
10	What is optimum pH value and optimum coagulant dosage of the coagulant process of your water sample.

Environmental Engineering Laboratory

Exp No: 7 Date:	Determination of available chlorine in Bleaching powder

Objective:

By this experimental study, the level of chlorine in bleaching is examined practically.

Aim:

To determine the percentage of available chlorine in the given sample of bleaching powder.

Theory:

Bleaching powder is used as a bleaching agent and also as a disinfectant. The main constituent of bleaching powder is calcium hypochlorite [$Ca(OCl)_2$] which supplies chlorine [Cl_2] with dilute acids.

$$Ca(OCl)_2 + 4HCl \longrightarrow CaCl_2 + 2H_2O + 2Cl_2$$

So the available chlorine is defined as the percentage of chlorine made available by bleaching powder when treated with dilute acids. The available chlorine present in bleaching powder sample is determined iodometricaliy by treating its solution with an excess of potassium iodide solution in the acidic medium.

$$ClO^- + 2H^+ + 2I^- \longrightarrow I_2 + H_2O + Cl^-$$

The liberated iodine (I_2) is treated with sodium thiosulphate ($Na_2S_2O_3$) solution using freshly prepared starch solution as indicator to be added near the end point.

$$[\text{ Starch} + I_2 \longrightarrow \text{Blue coloured complex }]$$

$$I2 + 2Na2S2O3 \longrightarrow Na2S4O6 + 2NaI$$

Apparatus required:

1) Analytical balance
2) Burette with Burette stand and porcelain tile
3) Pipette with elongated tips
4) Pipette bulb
5) Conical flask
6) Measuring flask
7) Funnel
8) Beakers

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Chemical required:

1) Bleaching powder
2) Standard sodium thiosulphate solution (N/10)
3) 10% Potassium iodide solution (KI)
4) Dilute acetic acid
5) Freshly prepared starch solution as indicator

Procedure:

- 1g of bleaching powder is accurately weighed in a weighing bottle and it is transferred to a clean beaker and is ground to a thin paste with water.
- The mixture is allowed to settle and the milky supernatant liquid poured into a 100 ml standard flask.
- The residue in the beaker is ground with a little more water and the operation repeated until the whole of the sample has been quantitatively transferred into the standard flask.
- Then the solution is made up to the volume. The flask is shaken well for uniform concentration.
- 10 ml of the bleaching powder solution (use measuring flask) in a state of very fine suspension is taken into a conical flask and add 10 ml of distilled water into it.
- Then add 10 ml of 10% potassium iodide (KI) solution and 10 ml of dilute acetic acid.
- This solution [which contains liberated iodine (I2)] titrates with standard sodium thiosulphate solution (taken in the burette) until the dark brown colour changes to pale yellow.
- To this add, 2 ml of freshly prepared starch solution as indicator, so the colour of the solution turned into blue. Continue the titration till the disappearance of blue colour of the solution and note down the volume of the titrant used.
- The titration is repeated until a concordant volume is obtained.

Tabulation:

Titration

Burette solution: Sodium thiosulphate

Pipette solution: Bleaching powder solution

Indicator: Starch

Environmental Engineering Laboratory

S.No	Volume of bleaching powder sample (ml)	Burette reading		Volume of Sodium thiosulphate consumed (ml)
		Initial (ml)	Final (ml)	
1				
2				
3				
Concordant Value (V_1)				

Normality of Standard $Na_2S_2O_3$ solution, $N_1 = 1/10 = 0.1$ N

Volume of Standard $Na_2S_2O_3$ solution, V_1 = _____ml

Volume of given bleaching powder solution, V_2 = _____ml

Normality of the given water sample, N_2

$N_1 X V_1 = N_2 X V_2$

Normality of given bleaching powder solution,

$N_2 = \frac{N_1 \times V_1}{V_2}$ = _____N

Amount of available chlorine = N_2 x Eq. Wt of Chlorine = N_2 x 35.45 g/ L

= _____g / L

Amount of available chlorine present in 100ml of the solution = _____ x 100 / 1000 g

= _____g

The percentage of available chlorine present in given sample of bleaching powder,

= (Amount of Chlorine / Weight of bleaching powder) x 100

= _____%

Result:

The percentage of available chlorine present in the given sample of bleaching powder is_____%

Inference:

Environmental Engineering Laboratory

Possible Viva-Voce

S.No	Query
1	What is disinfection?
2	Why do we prefer chlorination over other methods of disinfection?
3	Discuss the effect of pH of water and organic matter of water on efficiency of disinfection by chlorine.
4	What is Electro-katadyn process?
5	Why addition of chlorine tends to decrease the pH of water, while hypochlorite tends to increase the pH.
6	What are the different factors that affect the degree of disinfection?
7	What are different types of chlorine dosage process?
8	The dosage of chlorine should be proper. Why?
9	What is the acceptable limit of chlorine in drinking water?
10	In what form the chlorine is added to water for disinfection process?

Environmental Engineering Laboratory

Exp No: 8 Date:	Determination of Dissolved Oxygen

Objective:

By this experimental procedure, pollution strength of the sample is examined.

Aim:

To determine the dissolved oxygen content in the given water sample.

Theory:

The term Dissolved Oxygen (DO) is used to describe the amount of oxygen dissolved in a unit volume of water. DO is essential for the maintenance of healthy lakes and rivers. It is a measure of the ability of water to sustain aquatic life. The DO content of water is influenced by the source, raw water temperature, treatment and chemical or biological processes taking place in the distribution system. The presence of oxygen is good sign. Depletion of DO in water supplies can encourage the microbial reduction of nitrate to nitrite and sulphate to sulphide. It can also cause an increase in the concentration of ferrous iron in solution, with subsequent discoloration at the tap when the water is aerated. Hence, analysis of DO is an important step in water pollution control and wastewater treatment process control. There are various methods available to measure DO. In a healthy body of water such as lake, river or stream, the DO is about 8 parts per million. The minimum DO level of 4 to 5 mg/l or ppm is desirable for survival of aquatic life. Beyond certain point, most of the wastes break down. The concentration of DO rises as the river recovers oxygen from the atmosphere and aquatic plants. Thus DO test is the basis for BOD test which is an important parameter to evaluate organic pollution potential of a waste. It is necessary for all aerobic biological wastewater treatment processes to control the rate of aeration. DO can be measured either by titrimetric method or electrometric method.

Apparatus Required:

1) Burette with Burette stand and porcelain tile
2) Pipette with elongated tips
3) Pipette bulb
4) 500ml Conical flask
5) 300ml glass stoppered BOD bottles
6) 250ml graduated cylinders

7) Wash bottle

Chemical required:

1) Manganous sulphate solution
2) Alkaline iodide-azide solution
3) Concentrated sulphuric acid
4) Starch indicator solution
5) Sodium thiosulphate
6) Distilled or deionized water
7) Potassium Hydroxide
8) Potassium iodide
9) Sodium Azide

Procedure:

Preparation of reagents

Manganous Sulphate Solution

- Dissolve Manganese Sulphate (480g of $MnSO_4.4H_2O$ / 400g of $MnSO_4.4H_2O$ / 360g of $MnSO_4.4H_2O$) in freshly boiled and cooled distilled water, filter the solution and make up to 1000ml (1litre). In this experiment, we are using Manganese Sulphate Mono hydrate.
- Take 364g of Manganese Sulphate Mono hydrate ($MnSO_4.4H_2O$) and transfer it to the beaker. To dissolve the content, place it in the magnetic stirrer.
- The solution should not give blue colour by addition of acidified potassium iodide solution and starch.

Alkaline Iodide Sodium Azide Solution

- To prepare this reagent we are going to mix three different chemicals, dissolve either 500g of Sodium Hydroxide (or) 700g of Potassium Hydroxide and 135g of Sodium Iodide (or) 150g of Potassium Iodide.
- To prepare the reagent, take 700g of Potassium hydroxide and add 150g of Potassium iodide and dissolve it in freshly boiled and cooled water, and make up to 1000ml.
- Dissolve 10g of Sodium Azide (NaN_3) in 40ml of distilled water and add this with constant stirring to the cool alkaline iodide solution prepared.

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Sodium Thiosulphate stock solution

- Weigh approximately 25g of sodium thiosulphate ($Na_2S_2O_35H_2O$) and dissolve it in boiled distilled water and make up to 1000ml. Add 1g of Sodium Hydroxide to preserve it.

Starch indicator

- Weigh 2g of starch and dissolve in 100ml of hot distilled water. In case if you're going to preserve the starch indicator add 0.2g of Salicylic acid as preservative.

Testing of sample

- Take two 300ml glass stoppered BOD bottle and fill it with water sample to be tested. Avoid any kind of bubbling and trapping of air bubbles.
- Add 2ml of manganese sulphate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2ml of akali-iodide-azide reagent in the same manner.
- Squeeze the pipette slowly so no bubbles are introduced via the pipette. The pipette should be dipped inside the sample while adding the above two reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample.
- If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.
- Allow it to settle for sufficient time in order to react completely with oxygen.
- Add 2ml of concentrated sulphuric acid via a pipette held just above the surface of the sample.
- Carefully invert several times to dissolve the floc.
- At this point, the sample is "fixed" and can be stored for up to 8 hours if kept in cool, dark place.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure 203ml of the solution from the bottle and transfer to a conical flask.
- Tiration needs to be started immediately after the transfer of the contents to conical flask.
- Titrate it against sodium thiosulphate using starch as indicator. (Add 3 – 4 drops of starch indicator solution).
- End point of the titration is first disappearance of the blue colour to colourless.
- Note down the volume of sodium thiosulphate solution added which gives the DO.

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- Repeat the titration for concordant values.

Tabulation:

Titration

Burette solution: Sodium thiosulphate

Pipette solution: Tap water

Indicator: Starch

S.No	Temp (°C)	Volume of sample (ml)	Burette reading		Volume of Sodium thiosulphate (ml)	Dissolved Oxygen (mg/l)
			Initial (ml)	Final (ml)		
1						
2						
3						
Concordant Value						

Volume of Sample, =

Volume of Sodium thiosulphate, =

Normality of Sodium thiosulphate, =

Dissolved Oxygen, = ?

Dissolved Oxygen = Volume of Sodium thiosulphate * 0.2 * 1000 / Volume of water sample

=

Result:

The dissolved oxygen in the given water sample is _____mg/l

Inference:

Environmental Engineering Laboratory

Possible Viva-Voce

S.No	Query
1	Winkler titration method is based on _____ property of Dissolved Oxygen.
2	DO in water mainly depends upon?
3	The ingredients of Alkali are?
4	The precipitate formed after the addition of $MnSO_4$ and Alkali azide is?
5	Does DO depend only on physical properties of water?
6	Along the stream, the increase in DO will be at?
7	The DO in potable water imparts?
8	_____ and _____ interfere in the determination of DO.
9	The sample obtained for testing DO can be preserved by?
10	What is minimum DO for aquatic life survival in water?

Environmental Engineering Laboratory

Exp No: 9 Date:	Determination of Suspended, Volatile and Fixed solids

Objective:

By this experimental procedure, the quality of water sample and an approximate amount of organic matter present in the solid fraction of water sample is found.

Aim:

To determine the total Suspended, Volatile and fixed solids in the given water sample.

Theory:

The term total Suspended solids can be referred to materials which are not dissolved in water and are non filterable in nature. It is defined as residue upon evaporation of non filterable sample on a filter paper. The term Volatile solids refer to materials that are completely volatilized from water at higher temperature (550^{o}C). The solids are often referred to the organic content of the water. The term fixed solids can be referred to materials which are not volatilised from water at higher temperature (550^{o}C). These solids are often referred to the inorganic contents of the water. A well mixed sample is filtered through a weighed standard glass fiber filter and the residue retained on the filter is dried to a constant weigh at 103 – 105^{o}C. The increase in weight of the filter represents the total suspended solids. The sample is evaporated in a weighed dish on a steam bath and is dried to a constant mass in an oven at 103 – 105^{o}C. The reside obtained is ignited to constant weight at 550^{o}C. The remaining solids represent the total fixed solids and the weight lost during the ignition represents the total volatile solids.

Apparatus Required:

1) Evaporating Dish
2) Water bath
3) Hot Air Oven
4) Desiccators
5) Analytical balance
6) Measuring jars
7) Dish tongs
8) Crucibles
9) Filter

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10) Vacuum pump

11) Forceps (Smooth – Tipped)

12) Magnetic stirrer

13) Wash bottle

Chemical required:

1) Water sample

Procedure:

Testing of sample for Total Suspended Solids

- Place filtration apparatus with weighed filter in filter flask.
- Mix sample well and pour into a graduated cylinder to the selected volume.
- Apply suction to filter flask and seat filter with a small amount of distilled water.
- Pour selected volume into filtration apparatus.
- Draw sample through filter into filter flask.
- Rinse measuring jar into filtration apparatus with three successive 10ml portions of distilled water, allowing complete drainage between each rinsing.
- Continue suction for three minutes after filtration of final rinse is completed.
- Dry filter in an oven at 103 – 105^{0}C for at least 1 hour.
- Cool filter in desiccator to room temperature.
- When cool, weigh the filter and support.

Testing of sample for Total Volatile and fixed Solids

- To measure total volatile solids and fixed solids, take a clean silica crucible which has been washed and dried in a hot air oven at 105^{0}C for one hour and ignited at 550^{0}C to remove all organic materials present in it.
- Now weigh the empty silica crucible in analytical balance. Let's denote the weight measured as W1.
- Using pipette transfer 75ml of unfiltered sample in the porcelain dish.
- Switch on the oven and allowed to reach 105^{0}C. Check and regulate oven and furnace temperature frequently to maintain the desired temperature range.
- Place the silica crucible in the hot air oven and care should be taken to prevent splattering of sample during evaporation or boiling.
- Dry the sample to get constant mass. Drying for long duration is done to eliminate necessity of checking for constant mass.

- Cool the container in a desiccator. Desiccators are designed to provide an environment of standard dryness. This is maintained by the desiccant found inside. Don't leave the lid off for prolonged periods or the desiccant will soon be exhausted.
- We should weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.
- Samples need to be measured accurately, weighed carefully and dried and cooled completely.
- Note the weight of the residue as W2.
- Switch on the furnace and allow it to reach 550^{0}C. Check and regulate the furnace temperatures frequently to maintain the desired temperature range.
- Place the silica crucible in the furnace and care should be taken while keep the crucible inside the furnace since it will be too hot.
- Allow it to ignite for 20 minutes to get constant mass.
- As above, cool the silica crucible in a desiccator to room temperature.
- Weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.
- Note the weight with residue as W3.

Calculation:

Total Suspended solids

Weight of clean filter paper (g), W_1 =

Weight of clean filter paper and the residue (g), W_2 =

Weight of residue (g), W =

Volume of the sample (ml), V =

Total Suspended Solids (mg/l), TDS =

Weight of residue (g) = $W_2 - W_1$

Weight of residue (Convert g to mg) = W =

Volume of the sample (Convert ml to L) = V =

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Total Suspended Solids (mg/l) = W / V =

Total Volatile Solids

Weight of the clean silica crucible (g), W_1 =

Weight of the clean silica crucible and residue at 105^{0}C (g), W_2 =

Weight of the residue (g), W =

Weight of the clean silica crucible and the ash at 550^{0}C (g), W_3 =

Weight of the ash (g), W =

Volume of the sample (ml), V =

Total Volatile Solids (mg/l), TVS =

Weight of residue (g), $W = W_2 - W_3$

Weight of residue (Convert g to mg), W =

Volume of the sample (Convert ml to L), V =

Amount of total volatile solids present in the sample (mg/l) = W / V

Total Fixed Solids

Weight of the clean silica crucible (g), W_1 =

Weight of the clean silica crucible and residue at 105^{0}C (g), W_2 =

Weight of the residue (g), W =

Weight of the clean silica crucible and the ash at 550^{0}C (g), W_3 =

Weight of the ash (g), W =

Volume of the sample (ml), V =

Total Fixed Solids (mg/l), TFS =

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Weight of residue (g), $W = W_3 - W_1$

Weight of residue (Convert g to mg), W =

Volume of the sample (Convert ml to L), V =

Amount of total fixed solids present in the sample (mg/l) = W / V

Results:

In the given sample of water,

The Total Suspended Solids = _____ mg/l.

The Total Volatile Solids = _____ mg/l.

The Total Fixed Solids = _____ mg/l.

Inference:

Environmental Engineering Laboratory

Possible Viva-Voce

S.No	Query
1	The pore size of the filter paper used for filtration is?
2	Total suspended solids are mostly responsible for?
3	The chemical substance used in the desiccator is?
4	Always the total suspended solids value will be?
5	Total volatile solids determination is very important in the control of?
6	The crucible with sample, should be placed in the muffled furnace for atleast?
7	The total fixed solids is the measure of?
8	Putrescible solid means?
9	The total volatile solids is the measure of?
10	Why the crucible after ignition should be cooled in a desiccator?

Environmental Engineering Laboratory

Exp No: 10 Date:	Determination of Biochemical Oxygen Demand test

Objective:

By this experimental procedure, an idea on biodegradability of the sample is determined.

Aim:

To determine the biochemical oxygen demand in the given sample.

Theory:

The biochemical oxygen demand determination is a chemical procedure for determining the amount of dissolved oxygen needed by aerobic organisms in a water body to break the organic materials present in the given water sample at certain temperature over a specific period of time. BOD of water or polluted water is the amount of oxygen required for biological decomposition of dissolved organic matter to occur under standard condition at a standardized time and temperature. Usually, the time is taken as 5 days and the temperature is 20°C. The test measures the molecular oxygen utilized during a specific incubation period for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic material such as sulphides and ferrous ion. It also may measure the amount of oxygen used to oxidize reduced forms of nitrogen. The sample is filled in an airtight bottle and incubator at specific temperature for 5 days. The DO content of the sample is determined before and after five days of incubation at 20°C and the BOD is calculated from the difference between the initial and final DO. The initial DO is determined shortly after the dilution is made. All oxygen uptakes occurring after this measurement is included in the BOD measurement.

Apparatus Required:

1) BOD Incubator
2) Burette with Burette stand and porcelain tile
3) Pipette with elongated tips
4) Pipette bulb
5) 500ml Conical flask
6) 300ml glass stoppered BOD bottles
7) 250ml graduated cylinders
8) Wash bottle

Chemical required:

1) Calcium chloride
2) Magnesium Sulphate
3) Ferric Chloride
4) Di Potassium Hydrogen Phosphate
5) Potassium Di Hydrogen Phosphate
6) Di Sodium Hydrogen Phosphate
7) Ammonium Chloride
8) Manganous Sulphate
9) Potassium hydroxide
10) Potassium iodide
11) Sodium azide
12) Concentrated Sulphuric acid
13) Starch indicator
14) Sodium thiosulphate
15) Distilled or deionized water

Procedure:

Preparation of reagents

Manganous Sulphate Solution

- Dissolve Manganese Sulphate (480g of $MnSO_4.4H_2O$ / 400g of $MnSO_4.4H_2O$ / 360g of $MnSO_4.4H_2O$) in freshly boiled and cooled distilled water, filter the solution and make up to 1000ml (1litre). In this experiment, we are using Manganese Sulphate Mono hydrate.
- Take 364g of Manganese Sulphate Mono hydrate ($MnSO_4.4H_2O$) and transfer it to the beaker. To dissolve the content, place it in the magnetic stirrer.
- The solution should not give blue colour by addition of acidified potassium iodide solution and starch.

Alkaline Iodide Sodium Azide Solution

- To prepare this reagent we are going to mix three different chemicals, dissolve either 500g of Sodium Hydroxide (or) 700g of Potassium Hydroxide and 135g of Sodium Iodide (or) 150g of Potassium Iodide.

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- To prepare the reagent, take 700g of Potassium hydroxide and add 150g of Potassium iodide and dissolve it in freshly boiled and cooled water, and make up to 1000ml.
- Dissolve 10g of Sodium Azide (NaN_3) in 40ml of distilled water and add this with constant stirring to the cool alkaline iodide solution prepared.

Sodium Thiosulphate stock solution

- Weigh approximately 25g of sodium thiosulphate ($Na_2S_2O_35H_2O$) and dissolve it in boiled distilled water and make up to 1000ml. Add 1g of Sodium Hydroxide to preserve it.

Starch indicator

- Weigh 2g of starch and dissolve in 100ml of hot distilled water. In case if you're going to preserve the starch indicator add 0.2g of Salicylic acid as preservative.

Calcium Chloride Solution

- Weigh accurately 27.5g of anhydrous calcium chloride and dissolve it in distilled water.
- Take 100ml standard measuring flask place a funnel over it.
- Transfer it to the 100ml standard flask make up to 100ml using distilled water.

Magnesium Sulphate Solution

- Weigh accurately 22.5g of magnesium sulphate and dissolve it in distilled water.
- Take 100ml standard measuring flask place a funnel over it.
- Transfer it to the 100ml standard flask make up to 100ml using distilled water.

Ferric Chloride Solution

- Weigh accurately 0.15g of ferric chloride and dissolve it in distilled water.
- Take 100ml standard measuring flask place a funnel over it.
- Transfer it to the 100ml standard flask make up to 100ml using distilled water.

Phosphate buffer Solution

- Weigh accurately 8.5g of Potassium Di Hydrogen Phosphate (KH_2PO_4) and dissolve it in distilled water.
- Then add exactly 21.75g of Di Potassium Hydrogen Phosphate (K_2HPO_4) and dissolve it.
- To the same beaker 33.4g of Di sodium hydrogen phosphate ($Na_2HPO_4.7H_2O$), is weighed and added.

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- Finally to the beaker containing all the salts, add accurately 1.7g of Ammonium Chloride (NH_4Cl) and dissolve it.
- Take 100ml standard measuring flask place a funnel over it.
- Transfer it to the 100ml standard flask make up to 100ml using distilled water.
- The pH should be 7.2 without further adjustment.

Dilution water

- High quality organic free water must be used for dilution.
- The required volume of water (5 litres of organic free distilled water) is aerated with a supply of clean compressed air for at least 12 hours. Allow it to stabilize by incubating it at 20^{o}C for at least 4 hours.
- For the test we have taken 5 litres of organic free aerated distilled water, hence add 5ml each of the nutrients.
- Add 5ml of calcium chloride solution
- Add 5ml of magnesium sulphate solution
- Add 5ml of ferric chloride solution
- Add 5ml of phosphate buffer solution
- This is the standard dilution water. Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/l.

Testing of Sample

- Take four 300ml of glass stoppered BOD bottles (Two for sample & two for blank).
- Add 10ml of the sample to each of the two BOD bottles and fill the remaining quantity with the dilution of water i.e. we have diluted the sample 30 times.
- Add dilution water to the other two blank BOD bottles.
- After the addition of solutions, close the bottles and number it for identification.
- Now preserve one blank solution bottle and one sample solution bottle in the BOD incubator at 20^{o}C for five days.
- The other two bottles need to be analysed immediately.
- Avoid any kind of bubbling and trapping of air bubbles.
- Add 2ml of manganese sulphate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2ml of alkali-iodide-azide reagent in the same manner.
- Allow it to settle for sufficient time in order to react completely with oxygen.

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- When this floc has settled to the bottom, shake the contents thoroughly by turning it upside down.
- Add 2ml of concentrated sulphuric acid via a pipette held just above the surface of the sample.
- Carefully invert several times to dissolve the floc.
- Titration need to be started immediately after the transfer of the contents to conical flask.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure 203ml of the solution from the bottle and transfer to a conical flask.
- Titrate the solution with standard sodium thiosulphate solution until the yellow colour of liberated iodine is almost faded out.
- Add 1ml of starch solution.
- Note down the volume of sodium thiosulphate solution added, which gives the DO in mg/l. Repeat the titration for concordant values.
- After five days take out the bottles from the BOD incubator and analyse the samples and the blank for DO.
- Add 2ml of manganese sulphate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2ml of alkali-iodide-azide reagent in the same manner.
- If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.
- Allow it to settle for sufficient time in order to react completely with oxygen.
- Add 2ml of concentrated sulphuric acid via a pipette held just above the surface of the sample.
- Carefully invert several times to dissolve the floc.
- Titration need to be started immediately after the transfer of the contents to conical flask.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure 203ml of the solution from the bottle and transfer to a conical flask.
- Titrate the solution with standard sodium thiosulphate solution until the yellow colour of liberated iodine is almost faded out.

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- Add 1ml of starch solution and continue the titration until the blue colour disappears to colourless.
- Note down the volume of sodium thiosulphate solution added, which gives the DO in mg/l. Repeat the titration for concordant values.

Tabulation:

Titration

Burette solution: Sodium thiosulphate

Pipette solution: Sample

Indicator: Starch

Trial No	Day	Volume of sample (ml)	Burette reading		Volume of Sodium thiosulphate (ml)	Dissolved Oxygen (mg/l)
			Initial (ml)	Final (ml)		
Blank						
1						
2						
Sample						
1						
2						
Concordant Value						

Initial DO of the diluted sample, D_O =

DO at the end of 5 days for the diluted sample, D_5 =

Blank correction, B_C = $C_o - C_5$

B_C =

Initial DO of the blank, C_O =

DO at the end of 5 days for the blank, C_5 =

Biochemical oxygen demand = ({$D_O - D_5 - B_C$} x Volume of diluted sample) / Volume of sample

Result:

The BOD of the given sample of water is _____ mg/l.

Inference:

Environmental Engineering Laboratory

Possible Viva-Voce

S.No	Query
1	Biochemical Oxygen Demand is an important measure of?
2	In BOD test, the dilution water is aerated for?
3	What is the permissible limit of BOD in potable water?
4	Seeding the process of addition of?
5	What happens to the sample, after incubation period of BOD which is 5 days at $20^{o}C$?
6	In a treatment plant when the influent BOD is 245 mg/l and the effluent BOD is 22 mg/l, the percentage of BOD removed is?
7	The reaction that occurs between iodine and sodium thiosulphate result in?
8	Manganous hydroxide takes up dissolved oxygen in molecular form to form?
9	Sulphuric acid is added to reduce?
10	The increased level of BOD in water indicates?

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Exp No: 11 Date:	Determination of Chemical Oxygen Demand test

Objective:

By this experimental procedure, strength of the waste is assessed, which contain toxins and biologically resistant organic substances.

Aim:

To determine the chemical oxygen demand in the given sample.

Theory:

The chemical oxygen demand test is commonly used to indirectly measure the amount of organic compounds in water. Most applications of COD determine the amount of organic pollutants found in surface water, making COD a useful measure of water quality. It is expressed in milligrams per litre (mg/l), which indicates the mass of oxygen consumed per litre of solution. COD is the measure of the amount of oxygen in water consumed for chemical oxidation of pollutants. COD determines the quantity of oxygen required to oxidize the organic matter in water or waste water sample, under specific conditions of oxidizing agent, temperature and time. This method covers the determination of COD in ground and surface waters, domestic and industrial wastewater. The applicable range is 3 – 900 mg/l. The organic matter present in sample gets oxidized completely by potassium dichromate ($K_2Cr_2O_7$) in the presence of sulphuric acid (H_2SO_4), silver sulphate (AgSO4) and mercury sulphate (HgSO4) to produce CO_2 and H_2O. The sample is refluxed with a known amount of potassium dichromate ($K_2Cr_2O_7$) in the sulphuric acid medium and the excess potassium dichromate ($K_2Cr_2O_7$) is determined by titration against ferrous ammonium sulphate, using ferroin as an indicator. The dichromate consumed by the sample is equivalent to the amount of O_2 required to oxidize the organic matter.

Apparatus Required:

1) COD Digester
2) COD vials with stand
3) Burette with Burette stand and porcelain tile
4) Pipette with elongated tips
5) Pipette bulb
6) 250ml Conical flask

7) Tissue papers

8) Wash bottle

Chemical required:

1) Potassium dichromate

2) Sulphuric acid

3) Ferrous ammonium sulphate

4) Silver sulphate

5) Mercury sulphate

6) Ferroin indicator

7) Organic free distilled water

Procedure:

Preparation of reagents

Standard Potassium Dichromate reagent – Digestion solution

- Weigh accurately 4.913g of Potassium dichromate, previously dried at 103^{0}C for 2 – 4 hours and transfer it to a beaker.
- Weigh exactly 33.3g of mercuric sulphate and add to the same beaker. Measure accurately 167ml of concentrated sulphuric acid using clean dry measuring cylinder and transfer it to the beaker. Dissolve the contents and cool to room temperature.
- Take 1000ml standard measuring flask and place a funnel over it.
- Carefully transfer the contents to the 1000ml standard flask and make up to 1000ml using distilled water.
- This is the standard potassium dichromate solution to be used for digestion.

Sulphuric Acid Reagent – Catalyst solution

- Weigh accurately 5.5g silver sulphate crystals to a dry clean 1000ml beaker.
- To this carefully add about 500ml of concentration sulphuric acid and allow to stand for 24 hours.

Standard Ferrous Ammonium Sulphate Solution

- Weigh accurately 39.2g of ferrous ammonium sulphate crystals and dissolve it in distilled water.
- Take 1000ml standard measuring flask and place a funnel over it.
- Carefully transfer the contents to the 1000ml standard flask and make up to 1000ml using distilled water.

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Testing of sample

- Take three COD vials with stopper (Two for sample and two for blank solution).
- Add 2.5ml of the sample to each of the two COD vials and the remaining COD vials of for blank. To this COD vial add distilled water.
- Add 1.5ml of potassium dichromate reagent – digestion solution to each of the three COD vials.
- Add 3.5ml of sulphuric acid reagent – catalyst solution in the same manner.
- Cap tubes tightly. Switch on the COD digester and fix the temperature at 150^{o}C and set time at 2 hours.
- Place the COD vials into a block digester at 150^{o}C and heat for 2 hours.
- The digester automatically switches off. Then remove the vials and allow it to cool to the room temperature.
- Meanwhile, get ready with the burette for titration.
- Fill the burette with the ferrous ammonium sulphate solution, adjust to zero and fix the burette to the stand.
- Transfer the contents of the blank vial to conical flask.
- Add few drops of Ferroin indicator. The solution becomes bluish green in colour.
- Titrate it with the ferrous ammonium sulphate taken in the burette.
- End point of the titration is the appearance of the reddish brown colour.
- Note down the volume of ferrous ammonium sulphate solution added for the blank (A).
- Transfer the contents of the blank vial to conical flask.
- Add few drops of Ferroin indicator. The solution becomes green in colour.
- Titrate it with the ferrous ammonium sulphate taken in the burette.
- End point of the titration is the appearance of the reddish brown colour.
- Note down the volume of ferrous ammonium sulphate solution added for the sample (B).

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Tabulation:

Titration

Burette solution: Ferrous Ammonium Sulphate

Pipette solution: Sample

Indicator: Ferroin Indicator

S.No	Sample	Volume of sample (ml)	Burette reading		Volume of 0.1N FAS (ml)
			Initial (ml)	Final (ml)	
1	Blank 1				
2	Blank 2				
3	Sample 1				
4	Sample 2				
				Concordant Value	

Volume of ferrous ammonium sulphate for blank, (A) =

Volume of ferrous ammonium sulphate for Sample, (B) =

Normality of ferrous ammonium sulphate, N =

Volume of sample, V =

Chemical Oxygen Demand = (A – B * N * 8 * 1000) / Volume of sample

Result:

The COD of the given sample of water is _____ mg/l.

Inference:

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Possible Viva-Voce

S.No	Query
1	Potassium dichromate is considered as the best?
2	Mercury sulphate is added to reduce the interference of?
3	Silver sulphate is added as?
4	Ferroin indicator is?
5	After refluxing, _____ solution is titrated against FAS.
6	H_2SO_4 is added to FAS solution for?
7	The product formed after COD analysis is?
8	In industrial waste water, COD value is about _____ BOD value.
9	Sulphuric acid is added as?
10	A blank solution is?

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Exp No: 12 Date:	Determination of Residual Chlorine

Objective:

By this experimental procedure, chlorine dosage in domestic and industrial water is controlled which results in avoiding bad odour and change in the taste of water.

Aim:

To determine residual chlorine in the given water sample.

Theory:

Let us first understand the importance of adding chlorine. Treated or filtered water is deemed to be fit for consumption only if it is devoid of disease producing microorganism. Chlorine is primarily adopted to destroy or deactivate disease – producing microorganisms in the public water supplies and polluted rivers. Chlorine is usually added to water in gaseous form or as sodium or calcium hypochlorite. It has been practiced for several years. The word "Residual" means "remainder" or "that which is left", and as the name suggests the chlorine residual test is used to measure the amount of chlorine remaining in the water at the time the test is made. The Starch-Iodide titration method, one of the oldest methods of determining chlorine, is very non-specific for oxidants and generally is used for total chlorine testing at levels above 1mg/l Cl_2. Chlorine will liberate free iodine from potassium iodide (KI) solutions at pH 8 or less. The liberated iodide is titrated with a standard solution of sodium thiosulphate (Na2S2O3) with starch as the indicator. This method is based on the reaction with thiosulphate solution. The end point of the titration is indicated by the disappearance of the blue coloured, starch-iodide complex.

Apparatus Required:

1) Burette with Burette stand and porcelain tile
2) Pipette with elongated tips
3) Pipette bulb
4) 500ml Conical flask
5) Measuring jar
6) Wash bottle

Chemical required:

1) Concentrated Acetic acid (glacial)

2) Potassium Iodide crystals (KI)

3) Sodium thiosulphate

4) Starch indicator

5) Distilled or Deionized water

Procedure:

Preparation of reagents

Sodium Thiosulphate Solution (0.01N)

- Weigh approximately 2.482g of sodium thiosulphate ($Na_2S_2O_35H_2O$)
- Transfer to the beaker and dissolve it in boiled distilled water.
- Transfer it to the standard flask and make it up to 1000ml.

Testing of sample

- Rinse the burette with sodium thiosulphate and then fill the burette with sodium thiosulphate.
- Fix the burette to the stand.
- Take 200ml of a given sample in a conical flask.
- Add 5ml Acetic acid to acidify the sample. It is used to reduce the pH between 3 and 4 in the conical flask.
- Add about 1g Potassium Iodide (KI) measured using the spatula and dissolve it by thoroughly mixing it with stirring rod.
- Perform the titration quickly, since iodine liberated faster.
- Titrate the solution with standard Na2S2O3 solution until the yellow colour of liberated Iodine is almost faded out. (pale yellow colour)
- Add 1ml of starch solution and continue the titration until the blue colour disappears.
- In many cases residual chlorine is very low and starch needs to be added before starting up the titration.
- Note down the burette reading to know the volume of sodium thiosulphate added.

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Titration

Burette solution: Sodium thiosulphate

Pipette solution: Sample

Indicator: Starch

S.No	Temp (°C)	Volume of sample (ml)	Burette reading		Volume of Sodium thiosulphate (ml)	Residual Chlorine (mg/l)
			Initial (ml)	Final (ml)		
1						
2						
3						
Concordant Value						

Volume of Sodium thiosulphate, =

Normality of Sodium thiosulphate, = 0.01N

Volume of sample, =

Equivalent weight of chlorine, = 35.45

Residual chlorine = (Volume of Sodium thiosulphate * N * 35.45 * 1000) / Volume of sample

Residual chlorine (mg/l) =

Result:

The amount of residual chlorine in the given sample of water is _____ mg/l.

Inference:

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Possible Viva-Voce

S.No	Query
1	Disinfection means?
2	Bleaching powder is mixed in water for?
3	Residual chlorine means?
4	Potable water is?
5	The amount of residual chlorine in water should be?
6	Residual chlorine in water is detected by?
7	Chlorine is often added to wastewater for disinfection before effluent discharge. A potential problem with this procedure is?
8	Water requires _____ mg/l of chlorine to destroy the microorganisms present.
9	Chlorine is effective if the pH is?
10	Titration should be carried out with?

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